CODE IDENT NO. 25500

1063212

GOODYEAR AEROSPACE CORPORATION

AKRON 15, OHIO

RIGIDIZED INFLATABLE SOLAR ENERGY CONCENTRATORS

by

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6 Dec. 1963 9p

(NASA CR-53228; GER-11318 S1) OTS: ---

SECOND MONTHLY PROGRESS REPORT

COVERING PERIOD FROM 1 NOVEMBER THROUGH 30 NOVEMBER 1963

NASA CONTRACT NO. NAS1-3301, CONTROL NO. L-2923

December 6, 1963

Prepared for

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218-52 (5-63)
REF: ENGINEERING PROCEDURE S-017

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I. INTRODUCTION

This second monthly progress report covers the work accomplished from 1 November through 30 November 1963. The areas of investigation covered for this period include:

(1) Determination of physical properties of various materials.

(2) Investigation and construction of apparatus for screening tests.

(3) Investigation of reactions resulting from the blending of various materials.

Additional physical properties have been determined for various significant materials. An apparatus has been constructed to determine the "heat of decomposition" of the azide material. An azotometer is in the process of being assembled and will be used to determine the nitrogen release of the azide structures. A procedure has been resolved for the determination of the material sublimation tendency in high vacuum. The frothing character of prepolymer polyester resin, and the character of foam produced by combining polyoyl resin with an azide structure were also investigated. Progress in these areas are discussed below and the work expected to be accomplished during the next reporting period is presented. A program schedule is included, and indicates progress made to date.

The schedule forecast of manpower utilization, the schedule forecast of fund expenditures, and the actual cost figures are submitted separately in the "Monthly Financial and Manpower Utilization Report".

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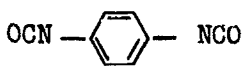
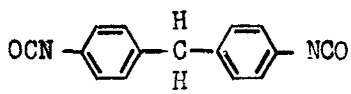
II. WORK ACCOMPLISHED DURING THIS REPORTING PERIOD

A. Experimental Materials

(Polyol resins were collected for evaluation as the resin component of precoat formulations. Preliminary experimentation has been done with one such, Voranol RS-375, a polyether octol with a heterocyclic nucleus. A source has been located for p-phenylene diisocyanate, the compound produced in situ by the decomposition of terephthaloyl azide (Azide Structure I)[1]. With this material it should be possible to study expeditiously certain aspects of the reactions between it and various polyols and look at physical properties of the resulting urethane polymers. A simple purification procedure has been found and applied to commercially available 4,4'-diphenyl methane diisocyanate to produce a purified material. This material is the same as that which will be produced in situ by decomposition of Azide Structure X^1 which is on order. It will find use in preliminary studies of the urethane producing reactions.

For future reference by structure the above mentioned diisocyanates are identified as follows:

STRUCTURE		NAME
I-1		p-Phenylene Diisocyanate (or 1,4-Benzene Diisocyanate)
X-1		4,4'-Diphenyl Methane Diisocyanate

Last month's effort to carry through a three step synthesis of 2,6-naphthalene diacyl azide has been scheduled to be continued at a later date. Further work is required to establish reasonable purity of the second intermediate product; an analytical procedure for this is in view.

B. Foaming Studies

Work has been largely directed at evaluation of Voranol RS-375 as the polyol resin component. Because of this material's octa-functionality and indications from the literature[2] that it yields urethane polymers with high heat distortion temperatures, there is some reason to hope that it will be useful. The following indications concerning it have been obtained:

1. Jouriles, N. and Welling, C. E.: Rigidized Inflatable Solar Energy Collectors First Monthly Progress Report, Akron, Ohio, Goodyear Engineering Report GER-11318, November 7, 1963.

2. I & EC Product Research and Development 2 (No. 3), 194 (1963)

1. It can readily be pre-polymered with tolylene diisocyanate (TDI) or Structure X-1 either with or without a tin catalyst.

2. In Forming the pre-polymer some care must be taken to have fast mixing to avoid local areas in which diisocyanate concentration is high enough to cause excessive cross-linking and gelation. The theoretical gelation point using diisocyanate is around -NCO/-OH = 0.38; we have not so far made a gel-free prepolymer with -NCO/-OH above 0.20.

3. There is promise that hi[illegible] PMT (polymer melt temperature on a temperature-gradient bar) can be obtained at low degrees of cross-linking with Voranol RS-375 than with PFR-6. This indicates the need for less azide in a formulation and hence a lesser azide exotherm problem. This is to be studied further and correlation of heat distortion temperature with PMT will be checked.

C. Measurement of Heat Release From Azide Rearrangement

(A test apparatus has been assembled and calibrated to determine the heat of decomposition of candidate azide materials.) The assembly is essentially a calorimeter which provides a means for controlling the initial or "triggering" temperature for the test material. The apparatus consists of a thin walled glass test tube enclosed in a glass dewar. Thermocouples placed in the test tube are connected to a chart recorder. The dewar is connected to a vacuum pump. The space between the test tube and the dewar inner wall is evacuated. The test tube surrounded by radiation shields (aluminized Mylar) and vacuum results in a low heat loss system.

Calibration of the system was accomplished by the method of mixtures. A solid specimen of known mass and specific heat was heated and lowered into the test tube containing an inert liquid (parafin oil). The equivalent specific heat of the system is calculated by use of the following equation

$$c_{cu}\, m_{cu}\, \Delta t_{cu} = c_s\, m_s\, \Delta t_s$$

(Heat lost by copper) = (Heat gained by system)

$$\text{or} \quad c_s = \frac{c_{cu}\, m_{cu}\, \Delta t_{cu}}{m_s\, \Delta t_s}$$

where: c_s = equivalent specific heat of calorimeter (cal/gm° C)

m_s = mass of inert fluid (grams)

Δt_s = temperature change of fluid (° C)

c_{cu} = specific heat of copper (cal/gm° C)

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m_{cu} = mass of copper specimen (grams)

Δt_{cu} = temperature change of copper specimen (° C)

The inert liquid (paraffin oil) was then heated and poured into the test tube. The cooling rate of the liquid was determined by the thermocouple and the recorder. When the temperature of the liquid dropped to the desired level, a copper specimen was lowered into the liquid. Temperature changes of the liquid and the copper specimen were recorded.

The determination of the heat of decomposition of the azide materials would be conducted basically the same as described for the calibration except that a known quantity of azide would supply the added heat to the inert liquid instead of the copper specimen.

D. Evaluation Methods for Azides

1. Preliminary study of standard methods for evaluation of shock and friction sensitivity has been made.

2. An azotometer[3] is under construction to permit studying thermal decomposition as a function of temperature and time.

3. A test fixture has been designed for measuring the sublimation tendencies of azides and polyol materials in high vacuum. Sublimation is to be determined by the rate of weight loss in vacuum. Measurements are to be made at various temperatures between 75 and 225° F.

4. A number of foam samples are produced in small volume and/or configurations unsuitable for density determination by the conventional method of weighing a large block. To accommodate such samples, a method of determining density has been developed that employs sand displacement to measure sample volume.

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3. A design is being adapted that is given by Siggia in Quantitative Organic Analysis Via Functional Groups, 3rd edition page 545.

III. AZIDE SUPPLIERS

Since Ash-Stevens Incorporated appears to be giving good service in the custom synthesizing of azides, little action has been taken to locate an alternate supplier. Two possible alternates that may fill this requirement are on file and will be contacted if needed.

IV. PROBLEM AREAS

Due to the time required to locate a supplier for the custom synthesized azides and to the time required for preparation and delivery of the first azide structure, Phase I of this program - the foam formulation - may require some extension of the date for selection of the optimum foam materials.

V. WORK TO BE PERFORMED DURING NEXT REPORTING PERIOD

A. Work to determine the utility of various polyols will be continued.

B. The measurement of chemical and physical properties of various azides will be continued as the evaluation methods are developed and as new azide structures become available.

C. Some material screening will be exercised, and some new azide structures will be designed.

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FIGURE 1

SCHEDULE - RIGIDIZED INFLATABLE SOLAR CONCENTRATOR INVESTIGATION